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**TESTING AND EVALUATION OF
PYROTECHNIC MIXTURES CONTAINING CHEMICALS**

by

Mitchell E. Penn

April 1970



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**DEPARTMENT OF THE ARMY
EDGEWOOD ARSENAL
Weapons Development and Engineering Laboratories
Ground Munitions Laboratory
Edgewood Arsenal, Maryland 21010**

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CONTAINING CHEMICALS

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Mitchell E. Penn

Flame and Pyrotechnic Systems Branch

April 1970

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Project 1B562602A081

DEPARTMENT OF THE ARMY
EDGEWOOD ARSENAL
Weapons Development and Engineering Laboratories
Ground Munitions Laboratory
Edgewood Arsenal, Maryland 21010

FOREWORD

The work described in this report was conducted under Project 1B562602A081, Chemical Dissemination/Dispersion Technology (U). This work is of a continuous nature and covers a period from about 1955 to the present.

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DIGEST

The methodology of testing pyrotechnic mixtures containing chemicals is explained. The use and the design of chambers, wind tunnels, and sampling equipment are described and analyzed.

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TESTING AND EVALUATION OF PYROTECHNIC MIXTURES CONTAINING CHEMICALS

I. INTRODUCTION.

This publication concerns methods currently in use at Edgewood Arsenal to test and evaluate the efficiency of pyrotechnic mixtures used to disseminate volatile solid chemicals, such as riot control chemicals—normally referred to as tear gas.

Because the aerosol produced is small and uniform, testing these pyrotechnic-based munitions is far easier than testing other methods of chemical dissemination. Pyrotechnic dissemination consists of heating and rapidly vaporizing chemicals inside a burning pyrotechnic munition. The chemical vapors exit through openings in the munition, rapidly cool, and condense to a smoke in the ambient air. The particle size of the chemicals in this smoke usually varies from 0.1μ to 1μ . Particles in this size range exhibit gas-like behavior in that their settling rate is extremely small, <0.5 cm/min. The other methods of disseminating chemical agents, such as explosive and cold-gas generation, tend to produce particles of extremely heterogeneous particle size, varying from several centimeters in diameter down to the micron range with the greatest amount being greater than 10μ and having falling rates of >0.5 m/min. Typical settling rates are shown in table I.

Since fallout of smoke particles is negligible, losses of chemical smoke generated from pyrotechnic mixtures and contained in an inclosure are minimal; these losses are a function of leakage, coagulation, impaction, and thermal and electrostatic precipitation effects.

II. MATERIALS, METHODS, AND DISCUSSION.

A. Test Chamber.

After consideration of the physics involved as well as results from other experimental programs, it was decided that chamber testing of chemicals was not only feasible, but also necessary. Accordingly, a cylindrical steel tank about 12 feet in diameter by 10 feet high with a volume of 45,000 liters was installed as a test chamber.

As shown in figure 1, the chamber was modified by installing a door, sampling ports, a scrubber-type exhaust system, and a mixing fan (in this case, a standard pedestal-type floor fan). The sampling ports consist of holes cut in the sides of the tank and closed by large rubber stoppers; the ports open into a glove box enclosure to cut down probability of contamination of nearby areas or operating personnel.

The testing method is as follows. The test device functions in the test chamber with the circulating fan in operation. The fan mixes the aerosol and the air in the tank until a homogeneous mixture is obtained. The time required to attain homogeneity is related to the size and shape of the tank and the mixing efficiency and location of the fan. This type of information was obtained experimentally when the chamber was first set up, by simultaneous chemical sampling at various locations and at selected time intervals after functioning a simple test munition. The time required to obtain homogeneity in the chamber was indicated by the uniformity of chemical test data obtained from various locations. For this chamber, 2 minutes was found to be an adequate mixing time. The loss rate of the typical aerosol tested in the chamber can be very low (figure 2).

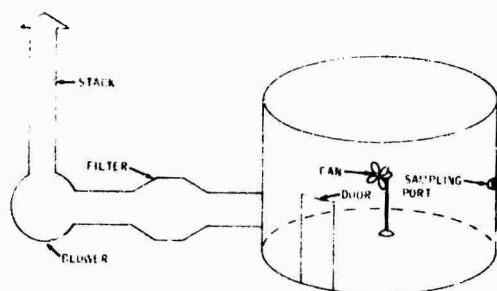


Figure 1. Test Chamber

Table I. Terminal Gravitational Settling

Diameter	Settling velocity
μ	<i>cm/min</i>
0.3	0.05
1	0.4
10	40
20	100

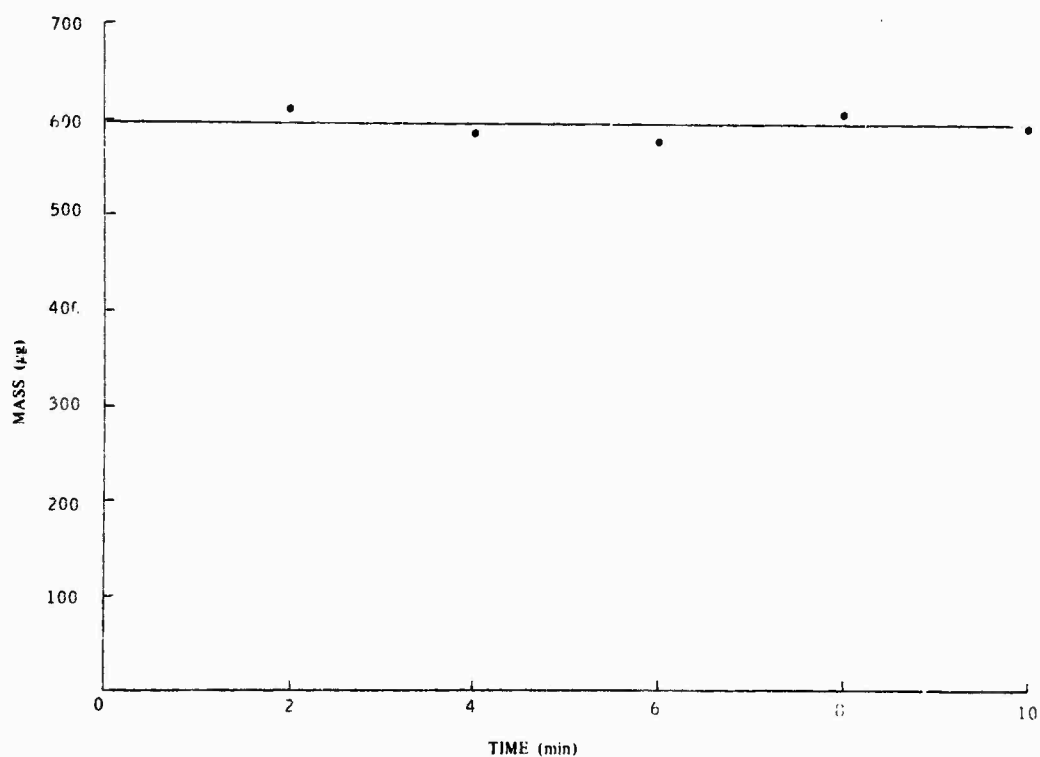


Figure 2. Loss Rate of Aerosol

The sampler used for most studies is glass-fiber filter paper specifically made for aerosol-filtration purposes and specially treated to remove all organic binder. This paper is tested to meet a minimum of 99.8% filtration efficiency for particles larger than 0.2 μ . Whenever fairly volatile materials are used, a bubbler is included to trap all gases from the filter paper exhaust stream. Flow rate is maintained by a critical orifice and a vacuum pump. The orifices are made of 1/4-inch brass tubing about 1.5 inches long. A flat disk is soldered to one end of the tube; a hole drilled through the center of the disk acts as a flat-edged orifice. As long as the pressure-drop ratio across this orifice is less than 0.53, the calibrated flow through the sampling system will be maintained. Each orifice is calibrated individually, and the orifice size is selected as shown in figure 3.

The typical sampling scheme takes four 1-liter samples at 1-minute intervals. After the test, each filter is carefully removed from its holder, placed in a sampling bottle, and submitted for chemical analysis. Typical tests provide 20- to 200- μ g samples, normally sufficient for analytical purpose. If a larger sample is required, a larger volume of air is sampled by using a higher flow orifice.

The analytical data obtained are plotted on a curve of mass versus time. The curve allows one to extrapolate back on a regression line of least squares to functioning time (time 0) and thus account for any losses inherent in the test facility. The method is illustrated in figure 2.

Because the sample obtained is an aliquot of about 1 to 45,000, the sample weight is multiplied by 45,000 to calculate the total weight of chemical contained within the test chamber, as follows:

$$\frac{\text{Sample weight (gm)}}{\text{Sampling time (minutes)}} \times \frac{\text{Chamber volume (liters)}}{\text{Sampling rate (liters/min)}} = \text{Total weight aerosolized}$$

One limitation to this test system is the concentration of chemical aerosol. Once this concentration rises above a certain value, loss rates increase exponentially due to radically higher coagulation rates. The upper limit for our chamber has been empirically set at about 100 gm of aerosolized chemical.

B. Chemical Sampling Tunnel.

Another device that may be used to test and evaluate the dissemination efficiency of these pyrotechnic devices is the chemical sampling tunnel, which looks like a wind tunnel (figure 4). In this method, the unit is functioned in the mixing section, sampled in the sampling section, and the exhaust scrubbed in the filter section. The critical conditions for proper operation of this system are accurate knowledge of air speed and homogeneity of the cloud. These conditions are met by using a large fan blade for mixing to obtain the necessary homogeneity and by making the velocity measurements in a modified entrance tunnel where flow is laminar.

Usual wind speeds range from 4 to 10 mph. A tunnel with a sample-section diameter of about 3 feet can sample all the munitions of current interest at Edgewood Arsenal. The limitation of agent weight is avoided in this device because this is a dynamic, not a static method.

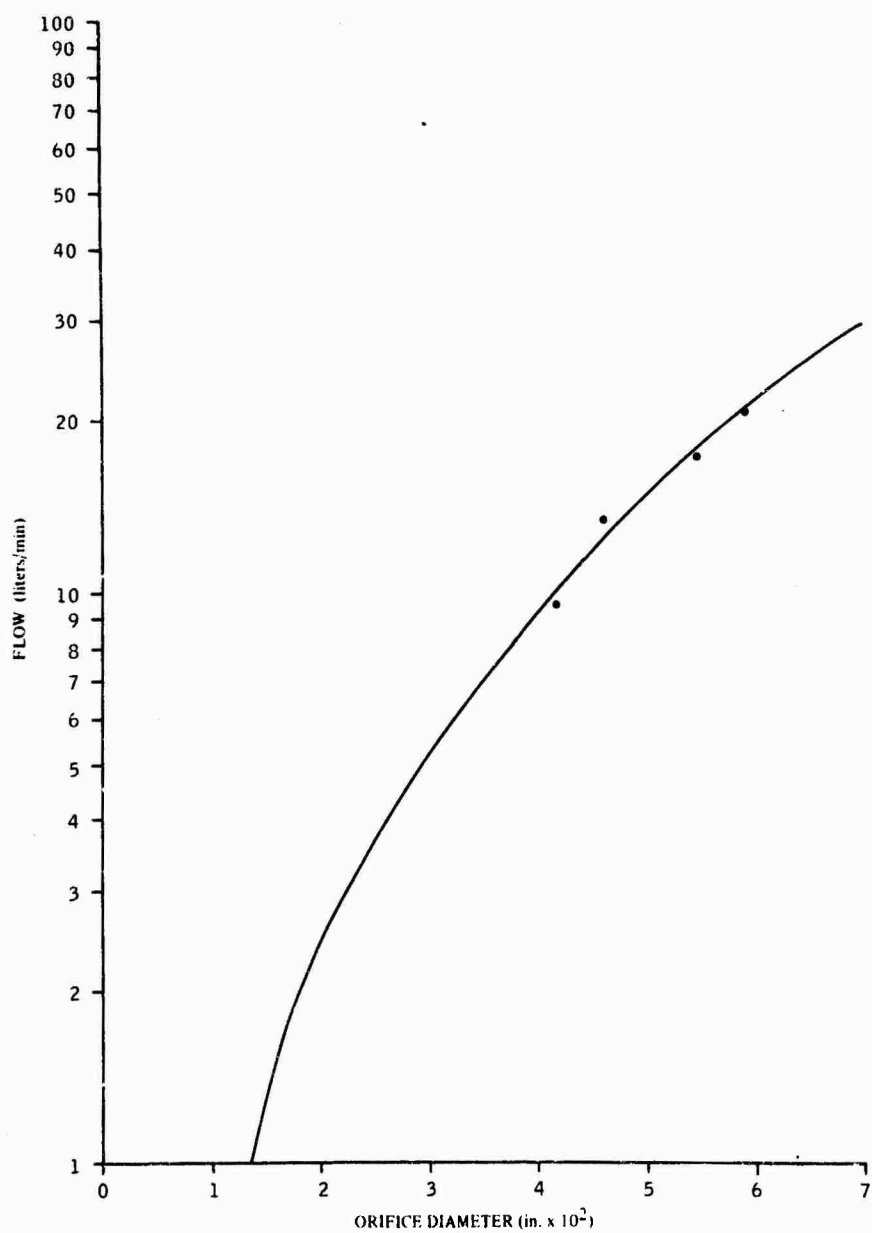


Figure 3. Flow Rate Versus Orifice Diameter

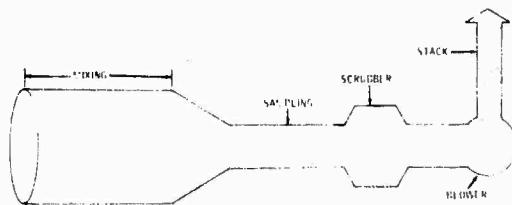


Figure 4. Chemical Sampling Tunnel

In this test design, the sampler is turned on, the munition is fired; and finally the sampling is stopped at some arbitrary time when there is no further visual evidence of aerosol in the tunnel. The sample represents a volume aliquot of all the volume which has passed through the tunnel; therefore, a tunnel factor that depends on air velocity is multiplied by the weight of chemical in the sampler to obtain the weight of chemical disseminated, as follows.

$$\frac{\text{Wind speed (cm/min)} \times \text{Sampling area (cm}^2\text{)} \times \text{Sample weight (gm)}}{\text{Sampling rate (cm}^3\text{/min)}} = \text{Weight aerosolized (gm)}$$

III. RESULTS AND CONCLUSION.

Testing results are normally reported in various munition-efficiency terms. The term most commonly used is "vaporization efficiency" how efficiently a chemical is vaporized from a pyrotechnic mixture. Another, perhaps much better term is "yield efficiency"—the amount of chemical aerosolized per weight of pyrotechnic mix. A final term, not often used, but the most descriptive, is "volume efficiency"—the amount of aerosol generated from any particular munition volume. A comparison of these terms, applied to imaginary mixtures, appears in table II.

Because most of the pyrotechnic munitions under consideration are low-density type and thus are volume, not weight, limited, the most important figure to a munitions-design engineer would be the last one, volume efficiency. This will allow him to calculate area-coverage factors obtainable from any particular munition system with a given volume available for pyrotechnic loading.

These test methods should be considered typical; but extensive alterations are frequently made to obtain various data.

Table II. Comparison of Efficiencies of Various Mixtures

Mixture *	Efficiencies		
	Vaporization (W_A/W_M) x 100	Yield (W_A/W_M) x 100	Volume [($\rho \times W_A$)/ V_M] x 100
	%		
I	100	10	10
II	80	40	40
III	80	40	60

* Mixture I: agent 10%, $\rho = 1$, gm aerosolized/10-gm mix = 1. Mixture II: agent 50%, $\rho = 1$, gm aerosolized/10-gm mix = 4. Mixture III: same as II, $\rho = 1.5$.

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